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## COMPOSITES AND SUBSAMPLES 5.1.1

Surface-water samples normally are composited and processed through sample splitting (subsampling) devices (NFM 2). Ground-water samples are not composited but are pumped either directly through a splitter or through a filtration assembly (filter assembly) into sample bottles, unless a bailer or other thief-type sampler is used to collect the sample. Inorganic-constituent samples usually are composited in the churn splitter, and organic-compound samples commonly either are composited in a 20-L glass, fluorocarbon polymer, or metal container, or are processed through a cone splitter.

**Only the Clean Hands person fills sample bottles with water withdrawn from the churn or cone splitter (NFM 4).**

Two types of water-sample splitters commonly used by the USGS are the polypropylene churn splitter (churn) and the fluorocarbon polymer cone splitter (cone).<sup>5</sup> Each splitter has specific advantages and disadvantages (NFM 2.2.1). By convention, the churn usually is used only for inorganic-constituent (and possibly for suspended organic carbon) samples. The churn is constructed of plastic materials that can potentially affect concentrations of other organic compounds. The cone is constructed of fluorocarbon polymer material and can be used for either inorganic-constituent or organic-compound samples. **Program or study protocols might dictate which equipment to use.**

- ▶ Either the churn or cone splitter can be used for splitting raw samples with suspended-sediment concentrations up to 1,000 mg/L.
- ▶ Only the cone splitter can be used for splitting raw samples with suspended-sediment concentrations up to 10,000 mg/L (Office of Water Quality Technical Memorandum 97.06).
- ▶ The splitting accuracy of the cone splitter is unknown for suspended-sediment concentrations between 10,000 to 100,000 mg/L (Office of Water Quality Technical Memorandum 97.06), but data are available that indicate the splitting accuracy of the cone is unacceptable at concentrations of 100,000 mg/L or more.

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### 5.1.1.A Churn-Splitter Procedure

Subsamples collected from the composite sample in a churn splitter must be processed according to the specific procedures described below, using Clean Hands/Dirty Hands (CH/DH) techniques as applicable.

1. Assemble sample-processing equipment and supplies on a clean work surface.
  - Put on appropriate, disposable, powderless gloves (gloves). (Wearing multiple pairs of gloves at one time provides an efficient means of changing gloves quickly.)

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<sup>5</sup>Consult the following references for more detailed information about the churn and cone splitters: Office of Water Quality Technical Memorandums 76.24-T, 80.17, 94.13, and 97.06; Capel and others (1995); and Capel and Larson (1996).

- If hand contact is made with a potential contaminant, remove the outer (contaminated) gloves before continuing with sample processing.
  - For CH/DH techniques: Remove churn splitter and inner bag from churn carrier. Leave the churn carrier and outer bag outside the processing area (vehicle or building).
2. Place all prelabeled wholewater or suspended-material bottles within easy reach of the churn spigot.
  3. Churn the composite sample at a uniform rate by raising and lowering the disk inside the churn splitter with smooth, even strokes.
    - When churning, the disk should touch bottom on every stroke, and the stroke length should be as long as possible without breaking water surface. **Do not break the surface of the water.**
    - **The churning rate should be about 9 inches per second (in/s).** If the churning rate is significantly greater than 9 in/s, or if the disk breaks the surface of the water, excessive air is introduced into the sample and could affect dissolved gases, bicarbonate, pH, and other characteristics of the sample.
    - Inadequate churning can result in withdrawal of nonrepresentative wholewater or suspended-material samples.
  4. Pre-mix the composite sample by churning for about 10 strokes to uniformly disperse suspended material before subsampling.
  5. **Raw subsample.** Withdraw the raw subsamples for wholewater or suspended-materials analyses first.
    - Withdraw an adequate volume of sample water for the field rinse while continuing to churn.
    - **Withdraw the first subsample.** The first subsample withdrawn from the churn should be the largest volume required (usually a 1-L sample).
    - Do not interrupt the churning/subsampling process, if possible. If an interruption occurs, reestablish the churning rate and remix the sample by churning ten strokes before resuming subsampling.
    - As the volume of composite sample in the churn decreases, adjust the stroke length to maintain a churning rate of about 9 in/s and avoid breaking the surface of the water being sampled.

6. Check requirements for sample preservation. **For raw samples that require chemical treatment → Go to section 5.4.**
  - For raw samples that require chilling without chemical treatment(s)—Pack samples in ice or refrigerate as quickly as possible. Maintain at or below 4°C without freezing (section 5.4).
  - For raw samples that do not require chilling or chemical treatment—Set samples aside in a clean area for shipping to the laboratory (section 5.5).
7. **Filtered samples → Go to section 5.2.** After wholewater or suspended-material subsampling is complete, use the remainder of the composite sample in the churn for filtered samples.
8. Empty the churn after the required number of samples has been processed.
  - If the churn will be reused during the field trip, disassemble and field clean onsite while still wet, as described in NFM 3.
  - If the churn will not be reused during that trip, rinse with DIW before it dries out, place it in a plastic bag and in the churn carrier to be transported back to the office laboratory for cleaning.
9. Document on field forms and in field notes the types of samples collected and the splitting procedures used.

**A field blank might be required after all sampling and processing equipment has been field cleaned (NFM 4.3).**

TECHNICAL NOTES: Subsamples totaling 10 L and 5 L can be withdrawn from the 14-L and 10-L churn, respectively, for samples for wholewater analysis. The sample volume remaining in either churn may be used for filtered samples.

The churn splitter is used to split samples with particle sizes  $\leq 250 \mu\text{m}$  and suspended-sediment concentrations  $\leq 1,000 \text{ mg/L}$ . Splitting accuracy becomes unacceptable at particle sizes  $> 250 \mu\text{m}$  and concentrations  $> 1,000 \text{ mg/L}$ .

### Cone-Splitter Procedure 5.1.1.B

Inorganic-constituent and organic-compound samples can be split using a fluorocarbon polymer (Teflon™) cone splitter. Although the cone splitter is used primarily for simultaneous distribution of surface-water samples into bottles, the cone also can be used similarly for a bailed or composited ground-water sample. The sample is poured into the splitter from the sampling device or transferred from a noncontaminating compositing container. If used for splitting pumped ground-water samples, the sample is pumped directly into the cone splitter.

1. Put on appropriate, disposable, powderless gloves (gloves). Remove cone splitter from protective covering.
2. Prepare a processing area that is protected from dust and fumes. Preferably, the cone splitter is installed in a processing chamber or covered with a large plastic bag.
3. Install cone splitter (see NFM 2, fig. 2-10, for a labeled diagram).  
**The cone splitter is built to close tolerances to achieve accurate and reliable operation and requires the following:**
  - **Use a bull's-eye level to level the cone splitter: this is critical for accurate performance.**
  - All tubes exiting the cone splitter must be the same length, as short as possible, and precleaned. Organic-compound samples require fluorocarbon polymer tubing. Carry a separate set of tubes for each site, and clean all sets on return to the office laboratory. If extra tubes are not available, do not reuse tubes for multiple sites without first cleaning them.
  - Push tubes as far as possible into the fittings on the splitter.

**Minimize atmospheric contamination—  
Cover the cone splitter and sample  
bottles during the sample splitting  
process and when not in use.**

4. Field rinse cone splitter and the appropriate sample bottles with the water to be sampled. **Do not field rinse laboratory-cleaned and baked glass bottles.**
  - a. Open cover to access cone-splitter reservoir. (Flap or access slots for hands can be cut into the plastic bag covering the splitter.)
  - b. Transfer 2 to 4 L of the sample into the cone-splitter reservoir. Some splitter reservoirs may be retrofitted with a funnel to ease pouring.
  - c. Close cover and lightly tap splitting system to dislodge adhering water drops. Discard rinse water.
  - d. Field rinse bottles for raw samples (RA, RU, and so on) with wholewater sample. Do not use the water sample previously processed through the cone splitter; follow directions in table 5-2.
5. Place bottles for raw samples under outlet tubes. Complete splitting procedure first with bottles for organic-compound samples, next with bottles for inorganic-constituent samples.
  - Place outlet tubes into sample bottles to prevent spilling. **Outlet tubes should not extend beyond the neck of the sample bottle. Do not submerge the ends of outlet tubes in the sample.**
  - Outlet tubes can be combined to collect various combinations of volumes of the original sample. Make sure no back pressure results from restrictions of water and air flow if combining outlet tubes into a single bottle.
  - Direct sample discharge from unused outlet tubes to waste.
6. Pour (or pump) sample into cone splitter. If hand contact is made with a potential contaminant while using CH/DH techniques, remove outer contaminated glove(s) or put on a new pair of gloves before transferring sample to cone splitter.
  - a. Gently shake or agitate sample for at least 10–15 seconds to resuspend any particulate matter present in sampler bottle or discrete sampler (such as a bailer).

- b. Transfer sample to cone-splitter reservoir (some splitter reservoirs may be retrofitted with a funnel to ease filling).
  - Open cone-splitter cover and invert sampler or compositor containing sample over splitter reservoir. (If using a bailer, empty through bottom-emptying device. If using a pump, hold sample line over the cone-splitter reservoir and pump sample directly into the cone splitter.)
  - First, collect organic-compound samples into clean, baked glass bottles (Appendix A5-A).
  - Next, collect inorganic-constituent samples into cleaned and field-rinsed polyethylene bottles or as designated (Appendix A5-B or A5-C).
- c. Maintain a head of water above the splitter standpipe to prevent air from entering the splitting block while rapidly transferring the sample. **Do not spill any of the sample when pouring or pumping it into the cone splitter.**
- d. For proper operation, the splitter standpipe must be discharging at full-flowing capacity.
  - **Never overfill sample bottle.**
  - **Always transfer the entire composite sample into cone splitter** for thorough distribution into the sample bottles.
7. **When splitting the samples, avoid exposing samples to direct sunlight or freezing conditions.** During sample splitting, the temperature of samples from the cone splitter should remain constant.
8. Close cone-splitter cover.
9. After flow has stopped, lightly tap the cone splitter to dislodge adhering drops.
10. Remove sample bottles and cap them immediately.
11. **To obtain smaller subsample volumes,** position bottles at cone outlet ports and pour a sample from the preceding set of split samples into the cone splitter. **For inorganics only, remember to rinse each new set of polyethylene sample bottles with DIW and sample as previously directed** (sections 5.0.1 and 5.0.3).

12. If multiple passes through the cone are required, randomize the ports selected. This minimizes bias from differences in ports caused by manufacturing processes.
13. Check requirements for sample preservation. **For samples that require chemical treatment → Go to section 5.4.**
  - For raw samples that require chilling without chemical treatment(s)—Pack samples in ice or refrigerate as quickly as possible. Maintain samples at or below 4°C without freezing (section 5.4).
  - For raw samples that do not require chilling or chemical treatment—Set samples aside in a clean area for shipping to the laboratory (section 5.5).
14. **Filtered samples → Go to section 5.2.** Remember to use only sample filtrate for the bottle field rinse.
15. Clean cone splitter, following instructions in NFM 3.
  - Disassemble and clean in the field before reusing. Field cleaning between sites must be done onsite while the cone splitter is still wet.
  - If the cone splitter will not be reused immediately, rinse with DIW and place in a plastic bag for transporting back to the office laboratory for cleaning.
16. Document on field forms and in field notes the types of samples collected and the splitting procedures used.

**A field blank might be required after sampling and processing equipment has been field cleaned (NFM 4.3).**